Report on Recommended Specifications for Microchemical Apparatus

Oxygen in Organic Compounds

Committee on Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society

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In previous reports (*5, 6, 9–20*) of the Committee on Microchemical Apparatus, recommended specifications were published for pieces of apparatus that were either the most widely used for the work in question or else an improvement over such apparatus according to tests made by the members of the committee or cooperating chemists. In this report, specifications are recommended for the direct microdetermination of oxygen in organic compounds via the water-gas reaction (*2, 7, 8, 21, 22*);

these specifications include the basic items used in both the gravimetric and volumetric procedures. (If necessary, the ends of the adjacent parts of *any* section of the apparatus may be equipped with the appropriate member of a S 12/5 ball and socket joint.)

CARRIER GAS

High-purity nitrogen is used as the carrier gas and may be purified further if necessary.

PURIFICATION UNIT

The purification unit shall consist of a preheater combustion furnace, a purification tube, and a drying and purifying tube.

(1) The preheater combustion furnace shall have a maximum over-all length of $4\frac{5}{16}$ inches (110 mm) with the thickness of the end plates not to exceed $\frac{1}{4}$ inch (6 mm). It shall accommodate combustion tubes up to 13 mm outside diameter. It shall be capable of continuous operation at temperatures up to 650°C and shall be provided with a holder which will permit attachment to a support rod. Several types of commercially available electric furnaces meet these requirements.

(2) The purification tube shall be similar in design to the Dumas nitrogen combustion tube with tip (*3, 8, 18*) with a minimum over-all length of 200 mm.

(3) The conventional bubble counter-U-tube (*4, 8, 18*) shall be used as a drying and purifying tube for the carrier gas.

PYROLYSIS UNIT

The pyrolysis unit shall consist of a long reaction furnace, a sample furnace, a reaction tube, a reaction tube closure, a carrier gas by-pass tube, members forming the carrier gas by-pass tube, and three-way stopcocks.

(1) Long Reaction Furnace

(a) The furnace shall have a maximum over-all length of 8 inches (203 mm) with the thickness of the end plates not to exceed $\frac{1}{4}$ inch (6 mm). The furnace shall accommodate reaction tubes up to 13 mm outside diameter. Electric heating elements shall be easily replaceable. The furnace shall be mounted on a substantial support which will permit a slight lateral displacement, if required.

(b) The furnace shall be capable of continuous operation at temperatures up to 1150°C. It must maintain a temperature of 1120° ± 15°C

over a length of 4 inches (102 mm) as measured inside the reaction tube. The temperature drop at the end adjacent to the sample furnace shall not exceed 150°C. Provision must also be made to obtain a minimum temperature of 1100°C at this point, either by lateral movement of the furnace or by means of auxiliary heating devices. Means shall be provided for regulating the temperature of the furnace.

(c) The furnace shall be equipped with a temperature idicator.

(d) An auxiliary heater, such as a ring gas burner (*1*), may be used.

(2) Sample Furnace

(a) The sample furnace shall have an over-all length not less than $1\frac{1}{2}$ inches (38 mm) or more than 4 inches (102 mm) with the thickness of the end plates not to exceed $\frac{1}{4}$ inch (6 mm). The furnace shall accommodate reaction tubes up to 13 mm outside diameter. Electric heating elements shall be easily replaceable. The furnace shall be mounted on a substantial support.

(b) The sample furnace must be capable of providing a temperature of 1150°C, as measured inside the reaction tube. The temperature drop from the center to a point $\frac{1}{2}$ inch (13 mm) from the end adjacent to the long furnace shall not exceed 150°C. Means shall be provided for regulating the temperature.

(c) The sample furnace shall be designed so that it may be either moved away from the reaction tube or turned off and cooled rapidly. In the latter case, the time for cooling to room temperature and reheating to operating temperature shall not exceed 30 minutes.

(d) If electric, the sample furnace shall be equipped with a temperature indicator.

(e) If a movable sample furnace is used, provision must be made for a minimum travel distance of $6\frac{1}{4}$ inches (159 mm) and for manual setting at any point. The rate of travel shall be between $\frac{1}{8}$ and $\frac{5}{8}$ inch (3–16 mm) per minute. An automatic control shall be provided to stop the sample furnace when it reaches the long furnace.

(3) Reaction Tube

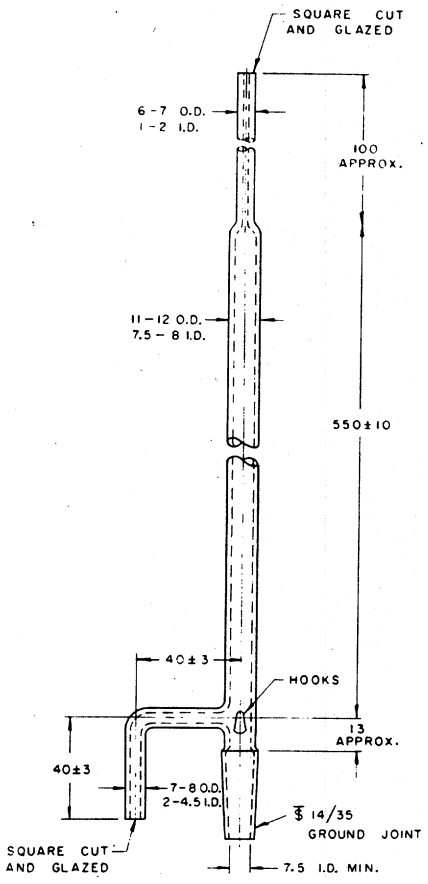
The reaction tube shall be made from the highest purity transparent quartz tubing. The design and dimensions shall be as shown in Fig. 1.

(4) Reaction Tube Closure

The reaction tube closure shall consist of a ground joint with stopcock (Fig. 1). It shall be made from borosilicate glass.

(5) Carrier Gas By-Pass Tube and Three-Way Stopcocks

The by-pass tube and stopcocks are used for backwash purposes; they cause a stream of nitrogen to flow backward through the tube in order to exclude air when a sample is being placed in the reaction tube. The tube may be made from metal, glass, or flexible tubing. The dimensions



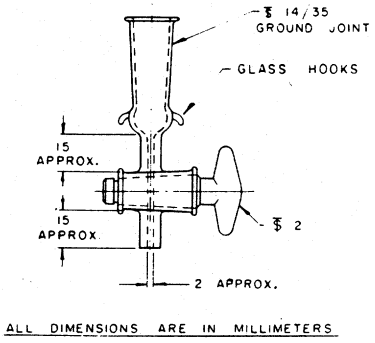


Fig. 1.

should conform with the rest of the apparatus. The by-pass tube is connected to the reaction tube by means of three-way stopcocks.

DESULFURIZATION UNIT (*7, 8*)

The desulfurization unit shall consist of an electric furnace and a desulfurization tube.

(1) Electric Furnace

(a) The electric furnace used for heating the desulfurization tube shall have a minimum over-all length of 4 inches (102 mm) with the thickness of the end plates not to exceed $\frac{1}{4}$ inch (6 mm). The furnace shall accommodate tubes up to 13 mm outside diameter. Electric heating elements shall be easily replaceable. Suitable means shall be provided to permit attachment to a support rod.

(b) The furnace shall be capable of continuous operation at temperatures up to 900°C as measured inside the desulfurization tube at the middle of the furnace. The temperature drop from the center to points $\frac{3}{4}$ inch (19 mm) for either end shall not exceed 100°C.

(2) Desulfurization Tube

The desulfurization tube shall be similar in design to the Dumas nitrogen combustion tube with tip (*3, 8, 18*) with a minimum over-all length of 200 mm, and shall be made from clear quartz.

ALKALI ABSORPTION TUBE

This tube shall conform to the dimensions shown in Fig. 2. The purpose of this tube is to absorb acid gases.

OXIDATION UNIT

The oxidation unit differs according to the procedure used to measure the end product.

(1) Gravimetric Procedure (*7, 8*)

The oxidation unit for the gravimetric procedure shall consist of a long stationary furnace, a copper oxide oxidation tube, an anhydrone drying tube, a carbon dioxide absorption tube, a guard tube, and a Mariotte bottle.

(a) The oxidation furnace shall conform to the same specifications as the preheater combustion furnace.

(b) The copper oxide oxidation tube shall be similar in design and

dimensions to the Dumas nitrogen combustion tube with tip (*3*, *8*, *18*) with a minimum over-all length of 200 mm.

(c) The anhydrone drying tube shall conform to the design and dimension of the absorption tube used in the microdetermination of carbon and hydrogen (*4*).

(d) The carbon dioxide absorption tube shall conform to the design and dimensions of the absorption tube used in the microdetermination of carbon and hydrogen (*4*). It should be made preferably of soda-lime glass.

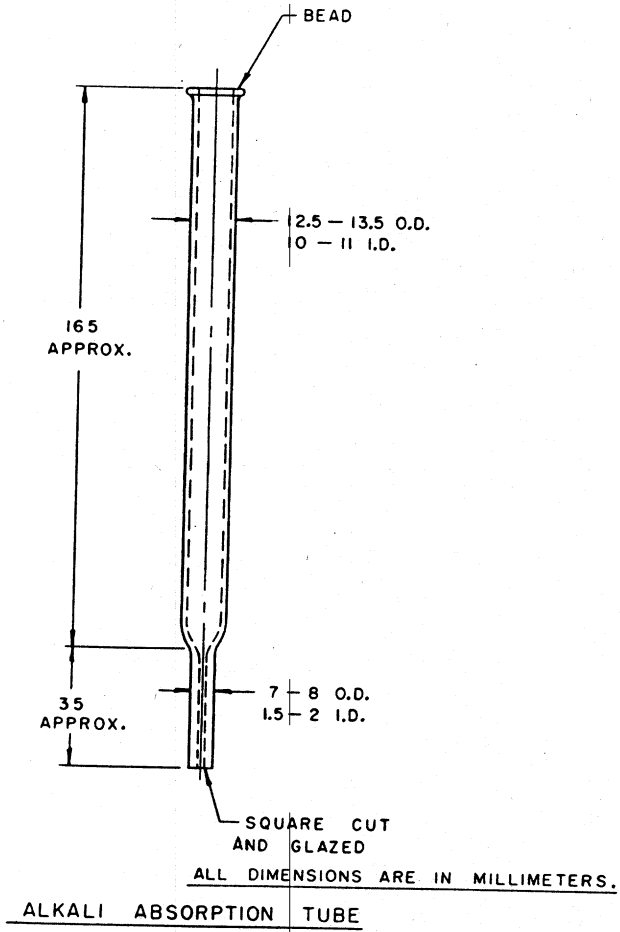
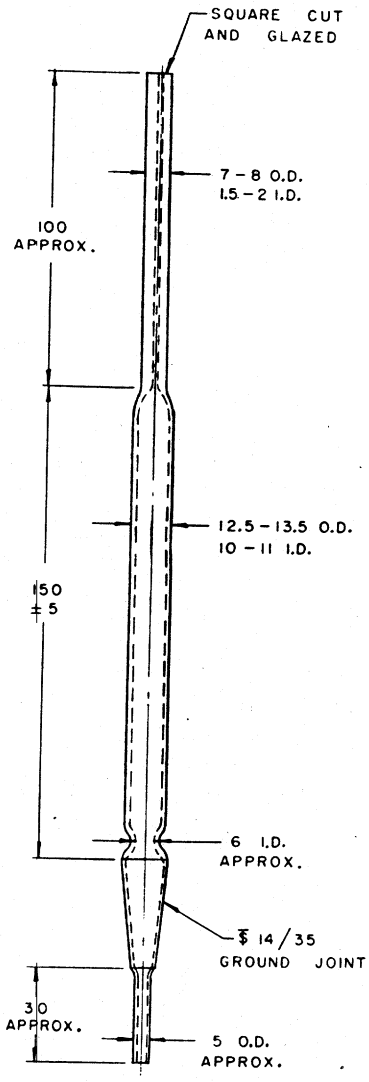


FIG. 2.



ALL DIMENSIONS ARE IN MILLIMETERS

IODINE PENTOXIDE OXIDATION TUBE

BOROSILICATE GLASS

FIG. 3.

(e) The guard tube shall conform to the design and dimensions of the guard tube used in the microdetermination of carbon and hydrogen (*4, 8, 18*).

(f) The Mariotte bottle shall conform to the design and dimensions

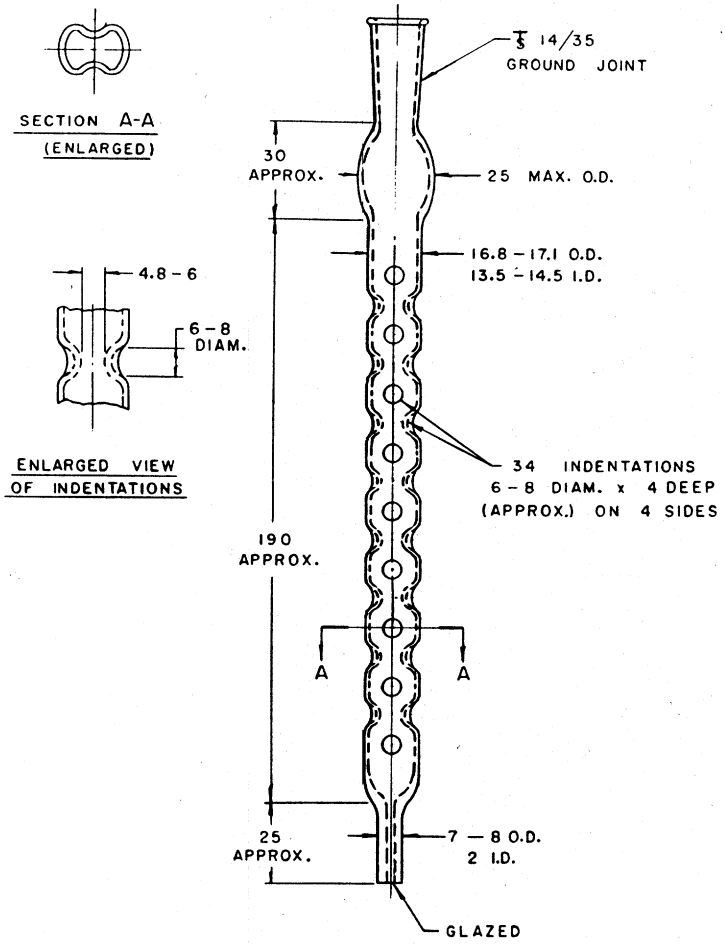


FIG. 4.

of the Mariotte bottle used in the microdetermination of carbon and hydrogen (*4*).

(2) Volumetric Procedure (*8, 21, 22*)

The oxidation unit for the volumetric procedure shall consist of an iodine pentoxide oxidation tube, a constant temperature block, an iodine absorption tube, and the Mariotte bottle described above.

(a) The iodine pentoxide oxidation tube shall be made to conform to the design and dimensions shown in Fig. 3.

(b) The constant temperature block shall have a maximum over-all length of 9 inches (230 mm) and shall accommodate tubes up to 15 mm outside diameter. At one end, the tube chamber of the block shall be enlarged to a diameter of $1\frac{1}{8}$ inches (29 mm) for a distance of $2\frac{1}{8}$ inches (54 mm) from the end.

The block shall be capable of continuous operation at temperatures up to 140°C. Means shall be provided for setting at any intermediate temperature and for maintaining this temperature to within $\pm$ 0.5°C.

If the block is mounted on a base, provision shall be made to permit vertical movement of the block so that the center of the heating chamber may be adjusted to any height from $8\frac{1}{2}$ to $9\frac{1}{2}$ inches above any surface used for support.

The block shall be provided with a temperature indicator and a pilot light.

(c) The iodine absorption tube shall be made to conform to the design and dimensions shown in Fig. 4.

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